

Crystallization by microwave energy: Effects on the survival probability of lithia-based glass ceramics

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Abstract: This study evaluated the survival probabilities of two lithia-based glass-ceramics after final crystallization in a microwave furnace using conventional crystallization as a reference. Disc-shaped samples of a lithium silicate (LS, Celtra Duo) and a lithium disilicate (LD, e.max CAD) were prepared and divided into two groups according to the crystallization method (n = 30): microwave (M) or conventional furnaces (C). The biaxial flexural strength test was used to determine the fatigue test profile and its parameters. Then, specimens were submitted to an accelerated life test (*step stress*) using three profile levels – mild, moderate, and aggressive – varying the load increments and the number of cycles until fracture (4 Hz). Survival data were used to calculate Weibull's beta (β) value and reliability of the assigned missions. Scanning electron microscopy was employed to analyze surface morphology, fracture characteristics, and failure patterns. Beta (β) values for the LS-C, LS-M, LD-C, and LD-M groups were 2.65, 0.25, 0.62, and 0.3, respectively. Similar reliability was observed in all groups after 50,000 cycles at 100 and 150 Mpa. At 200 Mpa, the crystallization method did not affect the reliability within LS or LD. However, LD showed greater reliability than LS when crystallized by microwave energy. Thus, microwave energy can be suggested as an alternative to the process of conventional lithia-based glass-ceramics crystallization without damaging their survival probabilities.

Keywords: Crystallization; Fractures, Stress; Microwaves.

Introduction

Dental glass-ceramics are materials in which their crystalline filler particles are precipitated within the starting glass nucleation and controlled growth heat treatments.^{1,2} Among these materials, lithium disilicate (LD) glass-ceramics became widely used because of the combination of excellent esthetics and improved strength (e.g., when compared with leucite-based glass-ceramics).³⁻⁵

Lithium disilicate crowns have shown high cumulative survival rates ranging from 86% to 96% in 10 years.^{6,7} Owing to these satisfying results, other lithia-based glass-ceramics have been developed, including lithium disilicate and/or lithium silicate crystals embedded in silicate glass.^{8,9} Follow-up studies of restorations made from these novel materials have



shown 97%–100% of survival in 1 year^{10,11} and 99% in 3 years.¹²

When available in blocks for milling in computer-aided design/computer-aided manufacturing (CAD/CAM), glass-ceramic restorations require a thermal treatment either before or after machining to reach their final crystallization. Crystallization firing leads to the final optical characteristics of the materials,¹³ enhancing the mechanical properties and decreasing the damage tendency of the materials.¹⁴

Previous studies have shown that microwave energy is an option to perform thermal treatment in dental ceramics.^{15,16} Electromagnetic waves may be reflected, absorbed, or transmitted, and this behavior is material-dependent.¹⁷ Electromagnetic waves increase the temperature to a point in which the restorative material starts absorbing a great part of the radiation, which accelerates the heating process and leads to the change in the crystalline phase of the materials. Studies have pointed out the finer microstructure and improved mechanical properties of microwave energy. Moreover, the volumetric heating (instead of centripetal heating) induced by microwave energy results in less energy consumption.^{17–19}

Even with the aforementioned advantages, the effect of glass-ceramics crystallization using microwave energy on the fatigue behavior of these materials is worrying. Ceramics are susceptible to degradation under the influence of mechanical, chemical, and/or biological stress (*i.e.*, fatigue).²⁰ Thus, the damage accumulated by cyclic forces in association with water molecules at the crack tip results in a chemically assisted slow crack growth. This process leads to material failure at stress levels lower than its fracture strength.^{21,22} In this sense, laboratory tests that reproduce in-service conditions are paramount to predict the clinical behavior of dental ceramics.

Given the above context, this study aimed to evaluate the survival probabilities of two lithia-based glass ceramics after final crystallization in a microwave furnace using the step-stress accelerated life testing (SSALT). The same materials were also fired in a conventional furnace and tested under the same conditions for comparison. The tested hypothesis

was that the firing method would not influence the reliability of the tested materials.

Methodology

Specimens preparation

Two lithia-based glass-ceramics were chosen for the study: LD-based (IPS e.max CAD, Ivoclar Vivadent, Schaan, Lichtenstein) and LS-based (Celtra Duo, Dentsply Sirona, York, USA) glass-ceramics. Blocks of both materials were milled into 12 mm-diameter cylinders using a lathe. Then, the cylinders were sliced into 1.2 mm-thick discs with a diamond blade in a precision cutting machine under water cooling (IsoMet 1000, Buehler, Lake Bluff, USA). The discs were polished on both sides with #400 grit silicon carbide papers in a polishing machine (Ecomet 250, Buehler, Lake Bluff, USA). Their top surfaces were finished with a sequence of #600, #1200, and #2500 silicon carbide papers. The polishing sequence was performed under water cooling, and a metallic device was used to ensure the discs were properly leveled.

The final discs of each lithia-based glass-ceramic were randomly divided into two subgroups ($n = 30$) according to final crystallization: conventional (LS-C and LD-C groups) or microwave (LS-M and LD-M groups) furnace. An industrial high-temperature microwave furnace was used to perform the crystallization cycles (FMO-1200, Fortelab, São Carlos, Brazil). A pilot study was conducted to determine the microwave crystallization settings for the M groups, as reported by Carvalho et al. (2020).¹⁶ The conventional and microwave crystallization cycles are described in Table 1. The C groups (conventional) were crystallized in furnaces from their manufacturers (IPS e.max CAD: Programat EP5000, Ivoclar Vivadent; Celtra Duo: Multimat, Dentsply Sirona, York, USA).

Surface characterization

Representative samples ($n = 2$) of each group were analyzed by scanning electron microscopy (SEM, Inspect S50, FEI Company, Czech Republic) to observe their surfaces according to crystallization methods. The ceramic discs were ultrasonically cleaned with

ethanol, dried, gold-sputtered, and inspected using a secondary electron detector at 25 kV.

Step-stress accelerated life testing (SSALT)

First, five samples were subjected to a monotonic flexural strength test to determine the fatigue profiles for the accelerated fatigue test. The piston-on-three-ball tests (ISO 6872/2015) were performed by placing the specimens over three support spheres (3.2 mm of diameter 120° apart, forming a 10 mm-diameter circle). The load was applied on the center of the ceramic discs at a 1 mm/min rate by a cylindrical flat piston (1.6 mm of diameter) fixed on a 1.000 kgF load cell until catastrophic fracture of the specimen. The tests were conducted in water using a universal testing

machine (Emic DL – 1000, Emic, São José dos Pinhais, Brazil). An adhesive tape was placed on the sample surface under compression to ensure better stress distribution and prevent fragment scattering. The flexural strength (σ) (MPa) was calculated according to Equation 1 (ISO6872/2015):

$$\sigma = \frac{-0.2387P(X-Y)}{b^2} \quad (1)$$

where P is the load (N), X and Y are parameters related to the material's elastic properties (Poisson's ratio and elastic modulus), and b is the specimen thickness at the fracture origin (mm).

After the monotonic tests, load profiles for the SSALT were drawn (Figure 1). Eighteen specimens

Table 1. Crystallization parameters for lithium disilicate (LD) and lithium silicate (LS) glass-ceramics according to different crystallization modes (conventional or microwave).

Material	Conventional		Microwave	
	LD	LS*	LD	LS
Initial temperature (°C)	403	500	0	0
Closing time (min)	6	1	8	8
Heating rate (°C/min)	90	60	5	5
Crystallization temperature (°C)	820	820	850	770
Maintenance time (min)	7	1	10	10
Opening temperature (°C)	700	750	300	300

*Since Celtra Duo does not require a final crystallization firing, the manufacturer recommended optional firing for increasing its flexural strength.

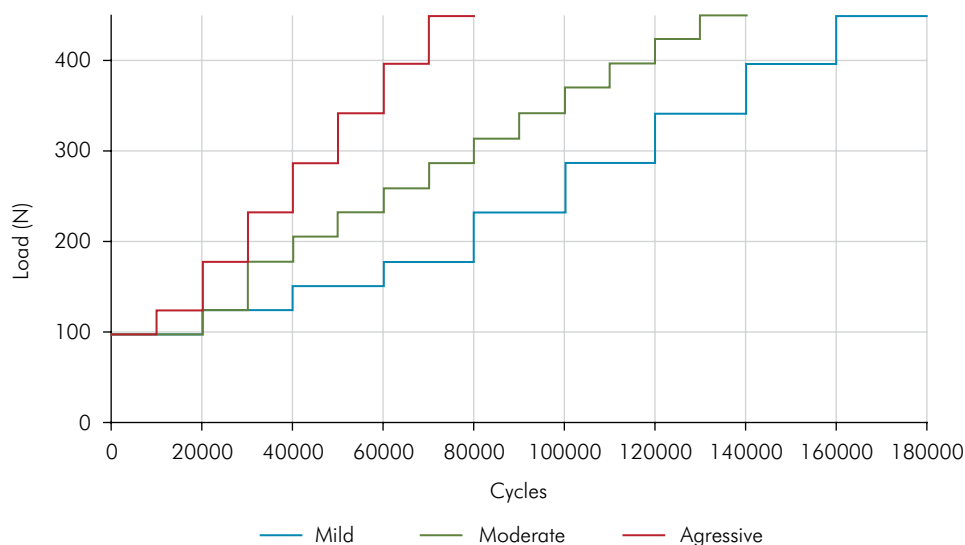


Figure 1. Load profiles (mild, moderate, and aggressive) used in the fatigue test by the step-stress method.

of each group were used in the proportion of 9:6:3 for each load profile (mild, moderate, and aggressive, respectively) as the methodology was tested in previous studies.^{23,24} The tests were performed in a fatigue-testing machine (Biocycle, Biopdi, São Carlos, Brazil) using the same test setup described for monotonic tests (piston-on-three-ball).

The loads were applied at a frequency of 4 Hz (as applied in previous studies^{24,25}) in distilled water until failure (fracture of the samples) or survival (no failure at the end of the step-stress profiles when the tests were suspended). When failure was detected, the load, step profile, and number of cycles were recorded. A maximum of 180,000 cycles were applied.

Fractographic analysis

To determine the fracture origin and characteristics, the fractured specimens were first evaluated in a stereomicroscope (Discovery V20, Zeiss, Jena, Germany). Representative specimens of each group were analyzed by SEM (Inspect S50, FEI Company, Brno, Czech Republic) to closely observe the fracture features (25 kV and secondary electron detector).

Statistical analysis

To describe the life data at different stress levels and the life–stress relationship, the results obtained from SSALT were analyzed using the Weibull ++ software (Reliasoft, Tucson, USA). Weibull distribution was chosen to fit the collected data. The Weibull analysis provides a beta (β) value that corresponds to the inclination of the regression line in a graph of probability and describes the behavior of the failure rate throughout time²⁶ as follows: $\beta < 1$, the failure rate diminishes throughout time, generally associated with “initial failure” or “inherent flaws”; $\beta \sim 1$, the failure rate does not vary over time, associated with random failure; and $\beta > 1$, the failure rate enhances over time, associated with failures related to damage accumulation. The differences between the experimental groups were evaluated by the absence of the overlapping of the confidence intervals to 90% of data reliability obtained by the two-sided Weibull statistics, according to the simulated missions.

Results

Surface characterization

SEM images revealed similar surfaces between the lithia-based glass-ceramics crystallized in conventional or microwave furnaces. Figure 2 shows similar features, such as grinding, and polishing marks.

Step stress accelerated life test

All specimens failed during the test. Beta (β) values for the LS-C, LS-M, LD-C, and LD-M groups were 2.65, 0.25, 0.62, and 0.3, respectively. The beta value of LS-C was significantly higher than that of LS-M. However, LS-C and LD-C showed comparable beta values, as well as LS-M and LD-M (Table 2). Figure 3 presents the use-level probability Weibull plots for each experimental group. These graphs illustrate the failure probability (unreliability) as a function of time (hours) for missions of 50,000 cycles at 100, 150, and 200 MPa.

Similar reliability was observed in all groups at 100 and 150 MPa (Table 2, Figure 3). At 200 MPa, the crystallization method did not affect the reliability within LS or LD. However, LD showed greater reliability than LS when crystallized by microwave energy. The opposite was observed when the materials were conventionally crystallized,

Table 2. Reliability for the missions of 50,000 cycles and different loads.

Reliability	LS-C	LS-M	LD-C	LD-M
Upper bound	0.99	0.98	1	1
100 MPa	0.97	0.94	0.99	0.99
Lower bound	0.87	0.84	0.94	0.97
Upper bound	0.97	0.91	0.98	0.99
150 MPa	0.9	0.81	0.95	0.97
Lower bound	0.67	0.64	0.84	0.91
Upper bound	0.91	0.67	0.91	0.95
200 MPa	0.7	0.49	0.8	0.89
Lower bound	0.28	0.29	0.59	0.74
Beta values	LS-C	LS-M	LD-C	LD-M
Upper bound	5.61	0.56	1.83	0.51
Beta	2.65	0.25	0.62	0.3
Lower bound	1.25	0.11	0.2	0.18

which both presented comparable reliability at 200 MPa.

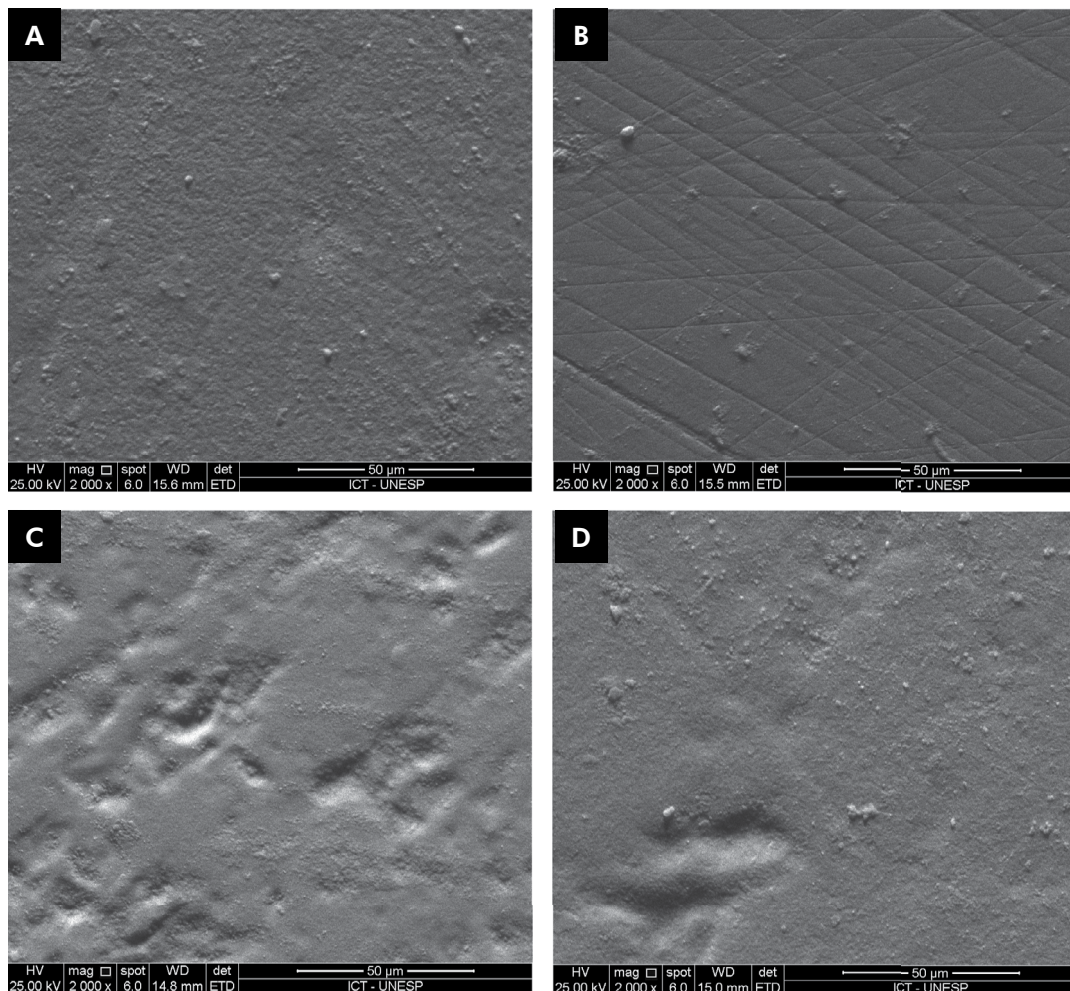
Fractographic analysis

Figure 4 shows the fractured samples of each experimental group tested under different stress profiles. As expected, all fracture origins were observed on the tensile side (i.e., opposite side of the load application). Lithium disilicate samples presented similar fracture features. Lithium silicate samples of both crystallization methods tested under the aggressive profile showed more pronounced fracture marks.

Discussion

Crystallization by microwave energy did not affect the reliability of either lithium silicate or lithium disilicate glass-ceramics. In addition, both materials showed similar surface and fractographic characteristics when crystallized by both methods. Hence, these main results led to the acceptance of our tested hypothesis.

These results do not indicate any difference regarding the microstructure of the ceramics crystallized in both methods. Polishing marks and grooves were observed in both microwave-crystallized



LD: lithium disilicate; LS: lithium silicate.

Figure 2. Scanning electron microscopy of the superficial morphology from different groups, at 2000 \times . A) LD crystallized by microwave energy. B) LD crystallized in the conventional furnace. C) LS crystallized by microwave energy. D) LS crystallized in the conventional furnace. Polishing marks are observed in all groups. LS specimens showed more surface grooves. However, similar surfaces are observed between microwave and conventionally crystallized specimens of each material (LD or LS).

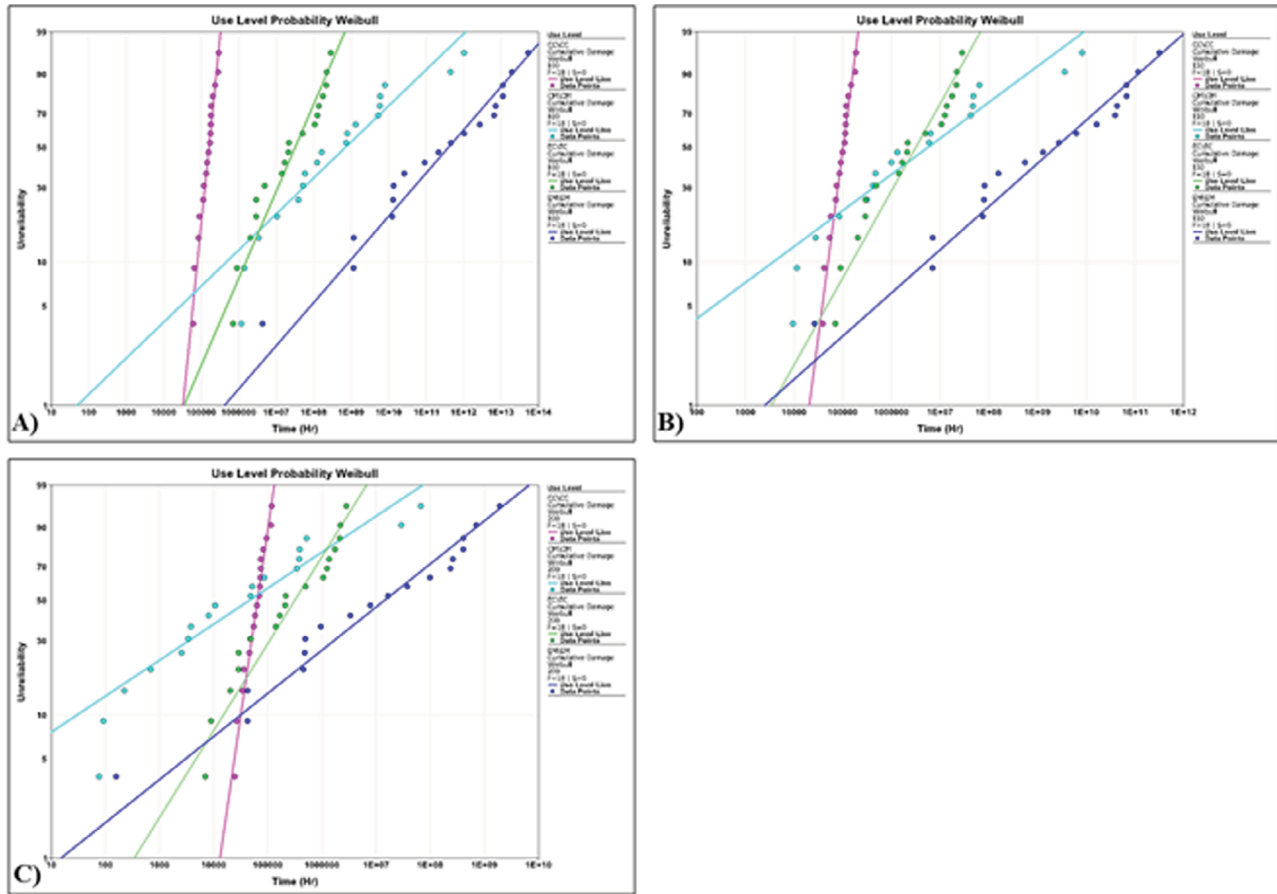


Figure 3. Weibull multiplots of failure probability according to number of cycles in different missions (90% CI): A) 100 MPa, B) 150 MPa, and C) 200 MPa. Similar reliability was observed among all groups at 100 and 150 MPa.

and conventionally crystallized materials (Figure 4). Similarly, Carvalho et al.¹⁶ demonstrated through SEM that microwave energy did not cause any harm to the microstructure of three glass-ceramics. However, Pendola et al.²⁷ showed an enhanced number of crystals as the consequent crystalline phase increased on LD specimens crystallized under hybrid heating in microwave.

Some advantages of the use of a microwave for thermal treatments of several materials can be highlighted, such as less energetic waste, better heating rates (with consequent decreasing of processing times), lower crystallization temperatures, and improvement in the mechanical properties of the material, specificity of energetic absorption, and technical simplicity.^{18,28,29} Nevertheless, Agrawal et al.²⁸ observed limited use of microwave

energy for ceramic materials because there could exist an inappropriate absorption of energy due to the physical phenomenon of reflection. Currently, this phenomenon does not occur at high temperatures, as in dental glass-ceramics crystallization. Thus, microwave energy can be effectively used in thermal processes involving metals and ceramics.¹⁹

Microwave energy is more deeply investigated as an alternative for sintering zirconium oxide dental ceramics.^{17,30,31} To the best of our knowledge, this is one of the first studies to attempt crystallizing commercial glass-ceramics with microwave energy and verify its effects on their mechanical properties. Li et al.³² developed approaches to affect crystal growth in a base glass network by microwave heating. The authors observed a denser microstructure in the microwave samples when compared with conventional firing.

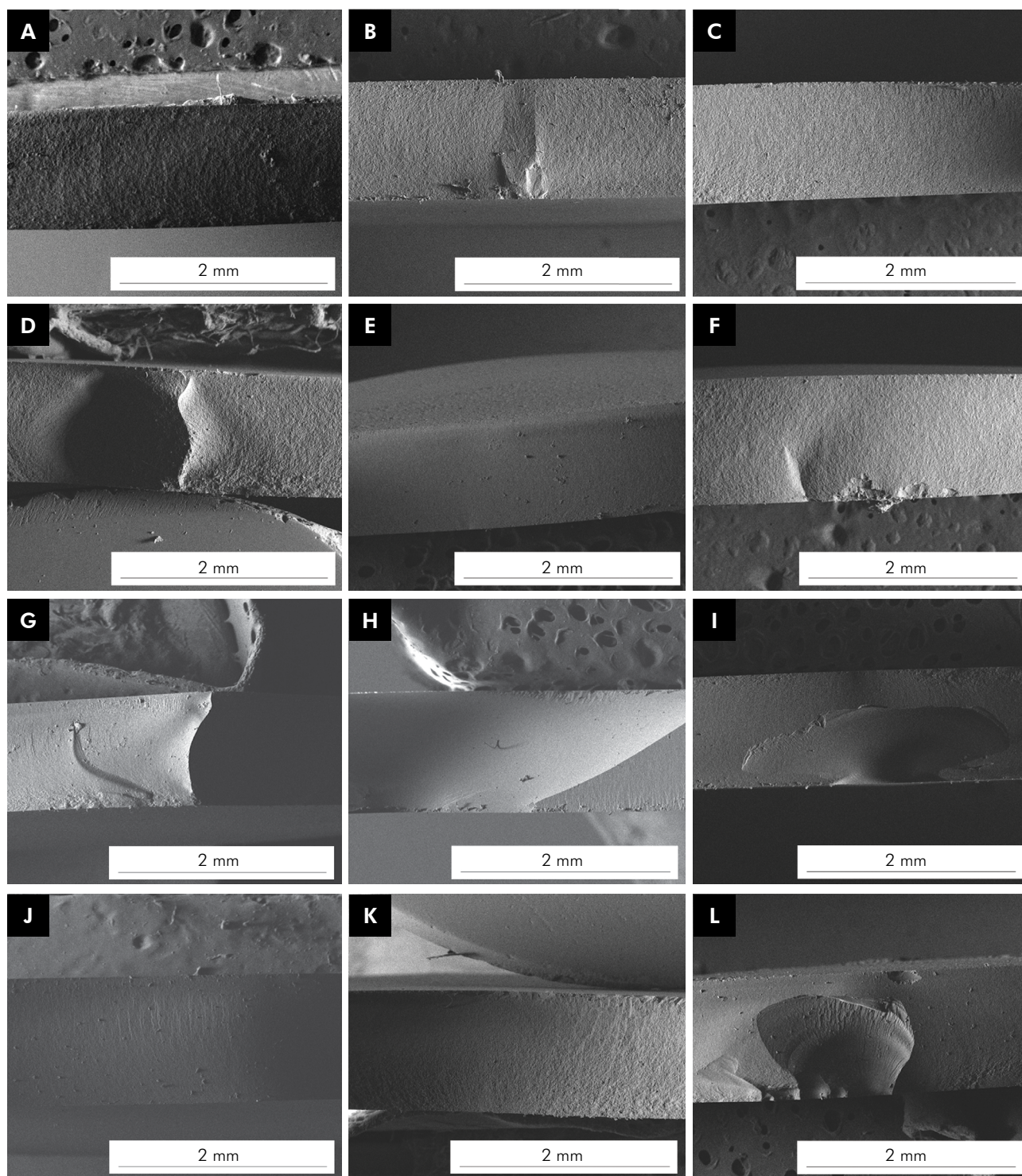


Figure 4. Scanning electron microscopy of a representative sample according to the group and profile used in the accelerated fatigue testing. A) Mild profile for the LD-C group, B) moderate profile for the LD-C group, C) aggressive profile for the LD-C group, D) mild profile for the LD-M group, E) moderate profile for the LD-M group, F) aggressive profile for the LD-M group, G) mild profile for the LS-C group, H) moderate profile for the LS-C group, I) aggressive profile for the LS-C group, J) mild profile for the LS-M group, K) moderate profile for the LS-M group, and L) aggressive profile for the LS-M group.

Carvalho et al.¹⁶ tested the same glass-ceramics used in our study. Their findings showed higher wear rates (three-body test) in the conventionally crystallized samples than in microwave crystallization regardless of the ceramic material. Together with our results, this reveals that microwave energy is a promising alternative for glass-ceramics crystallization. Hence, more studies on this topic are warranted to ensure that more ceramic materials can be crystallized in microwave furnaces without jeopardizing their properties.

Our fatigue results showed a better performance of the LD-M group, which presented 4% less failure probability than LS-M after 50,000 cycles at 200 MPa. The better performance of the lithium disilicate groups than the tested lithium silicate glass-ceramic is in agreement with the results of previous studies.³³⁻³⁶ The superior mechanical behavior of LD is attributed to the needle-shape crystals, which corresponds to 70% of the material volume. In addition, compression stress surrounding the crystals collaborates for a crack deflection, and a small amount of glass matrix improves its properties in the presence of fatigue, providing the material with appropriate toughness and strength.¹⁴

However, this difference was not observed when the glass-ceramics were conventionally crystallized. The LS glass-ceramics tested in our study (Celtra Duo) does not require a crystallization firing because it can be used as a chair-side material. Nonetheless, the manufacturer claims that optional firing can improve its flexural strength in ~370 MPa (manufacturer instructions). As additional firing was performed,

it appeared to have affected the failure probability of the material, making it comparable to LD. Notably, 1 LS-C was the only group that showed $\beta > 1$, which is associated with damage accumulation, whereas other groups showed $\beta < 1$, which indicates failures due to inherent internal flaws. These findings point out the benefits in the mechanical behavior of Celtra Duo brought by additional firing, which apparently was not produced by microwave energy, because the reliability of LD-M and LS-M was similar and LS-M showed $\beta < 1$.

Despite the encouraging aforementioned reliability results, the absence of pH variations, temperature, and sliding load figures are some limitations of this study. In addition, further fatigue tests with single-crown samples and different antagonists are needed to understand the survival behavior of microwave-crystallized restorations in more realistic testing setups. Even so, this study suggests that microwave energy can be an alternative for glass-ceramics crystallization in the future.

Conclusion

Crystallization by microwave energy produced lithium silicate and lithium disilicate samples with similar survival probabilities when compared with their conventional crystallization counterparts. Owing to these results, microwave energy can be suggested as an alternative to conventional glass-ceramics crystallization without harming the reliability of these materials.

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